

# STUDY OF MICROSTRUCTURAL FACTORS INFLUENCING SURFACE MORPHOLOGY OF Ti THIN-FILMS

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Sandia National Laboratories (SNL) has active programs in developing microfabrication technology. One of the technologies of interest is X-ray lithography, electrodeposition, and molding (LIGA). In the LIGA process, a multi-layer Ti-Cu-Ti thin-film composite on Si wafer is used as a supporting substrate for poly methylmethacrylate (PMMA) photo resist molding. The two Ti films provide adhesion for the interfaces between Cu-PMMA at the top and Cu-Si wafer at the bottom. During our process development, we encountered an adhesion-related problem mainly at the Ti-PMMA interface. Since good adhesion of PMMA on the top Ti film surface is critical for LIGA's success, we have undertaken research to study process-related and material-related factors that affect adhesion of PMMA on Ti. One of the known variables affecting adhesion is surface roughness because of its effect on wetting and impurity trapping at bonding surfaces. In general, there is an optimum surface roughness. Roughness values above or below the optimum could be harmful to adhesion. To gain a better understanding of the optimum surface roughness for our composites, we have studied the effect of composite grain structure in conjunction with surface treatments such as surface etching. In this presentation we will discuss the difference in grain structure of Cu films fabricated by different deposition processes. In addition, the effect of the underlying Cu microstructure on the surface morphology and roughness of the Ti film will be discussed.

Construction of the thin-film Ti-Cu-Ti composite is shown in figure 1. The nominal thicknesses of Ti, Cu, and PMMA films are 50nm, 300nm and 1.2mm respectively. Two Ti-Cu-Ti composites were investigated. One made by Ar-plasma sputtered deposition (PVD) and the other by thermal vapor deposition (TVD). Surface morphology and roughness were examined using field emission scanning electron microscopy analysis (FESEM) of the composite surfaces prior to the PMMA coating. The microstructures of composites were examined by transmission electron microscopy (TEM). The atomistic structure of the interface between Cu and Ti was studied using high resolution transmission electron microscopy (HRTEM). Specimens for TEM and HRTEM analyses were thin film cross-sections prepared by an ion milling technique.

For the PVD composite, FESEM showed features on the surface of the upper Ti film which are ~50-100nm in size (Fig 2a). These features are relatively rough compared with the Ti surface on the flat single crystal Si wafer (Fig 2b). TEM images of the PVD thin-film cross-section show that the construction and film thicknesses of the composite are as expected (Fig 3a). The overall Ti top surface appeared to be curved (Fig. 3a). The curve length is about 50-100nm, which is similar to the features seen on this surface by FESEM. The Cu film is ~400nm thick. The Cu grains appeared to be columnar (high aspect ratio) with the grain width varying through the film thickness, increasing from bottom (Si side) to top (PMMA side). The average width of columnar Cu grains at the top is ~ 50-100nm. High magnification TEM shows that the size and curvature of Ti surface features almost directly resemble the morphology of the Cu grains underneath (Fig. 4a), i.e. the Ti grain size and surface curvature correspond to those of the Cu grains underneath. For the TVD composite, the construction and film thicknesses of the composite are also as expected (Fig. 3b). However, The overall top Ti surface is relatively smooth as compared with the PVD film. The Cu grains are not columnar and the overall surface is much flatter. The average Cu grain size is much larger than in the PVD film. In some cases the grain size is 200-300nm, which is as large as the Cu film-thickness. There is little evidence of grain size variation through the thickness as was seen in the PVD Cu film. High magnification TEM shows that the top Ti grain structure, size and surface morphology do not correspond to those of the Cu grains underneath (Fig. 4b). The smaller Ti grains appear to nucleate randomly on the flat, large-grain Cu surface.

The above results indicate that the Cu grain structure is quite different between the two composites made by PVD and TVD. We are interested in knowing whether this microstructural difference causes any difference in orientation relationship between Cu and Ti grains during nucleation at the interface. HRTEM imaging shows that most Ti grains grown on both PVD- and TVD- Cu have random orientation relative to the Cu substrate, i.e. there is no noticeable difference in Ti grain orientation that is attributable to the microstructure difference in underlying Cu films. For both composites, we occasionally observed close

pack plane alignment, i.e.  $\text{Ti}(1, 0, -1, 0) // \text{Cu}(111)$  at the top Ti-Cu interface (Fig 5). In the HRTEM images, we also observed an  $\sim 5$  nm-thick surface oxide layer, presumably  $\text{TiO}_2$ , on the top Ti layer in both composites (Fig. 6). This ultra thin surface oxide did not appear to alter the surface curvature or roughness of the Ti layer.

These SEM, TEM, and HRTEM results suggest that grain structure of the Cu substrate is highly dependent on the deposition process. PVD produced fine columnar Cu grains with a curved top

surface, whereas TVD produced large Cu grains with a flat top surface. The results also suggest that the Cu grain structure, size and surface morphology, in particular, are largely responsible for the Ti surface morphology and roughness. This means that the deposition process for the thin film composite is an important criteria for optimizing surface roughness in the overall LIGA process. Studies of the effect of various deposition processes on Cu grain nucleation and growth, and optimum titanium roughness are in progress.

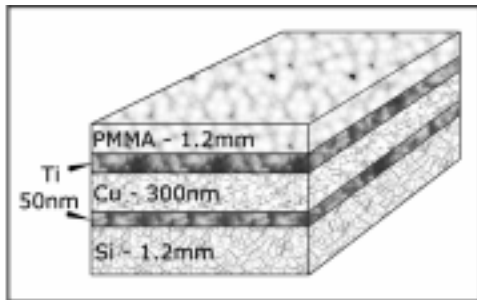


Figure 1

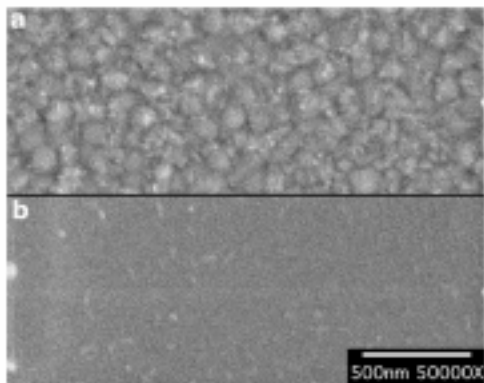


Figure 2

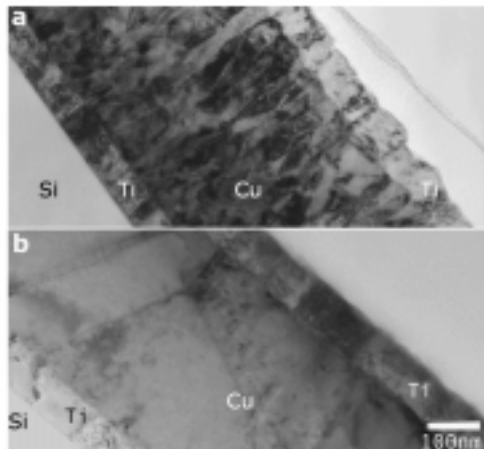


Figure 3

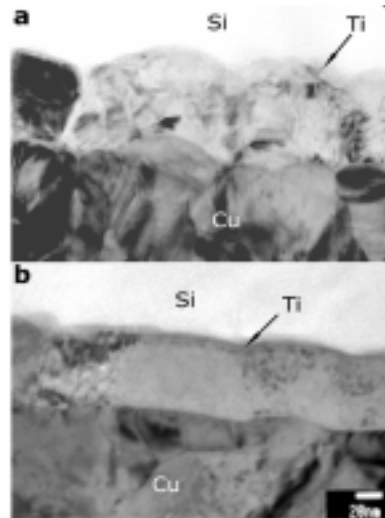


Figure 4

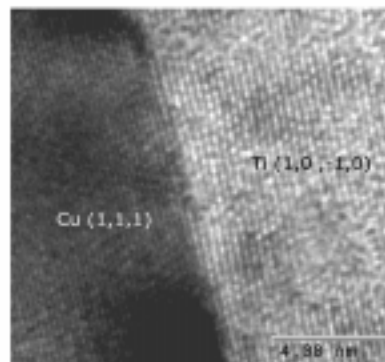


Figure 5

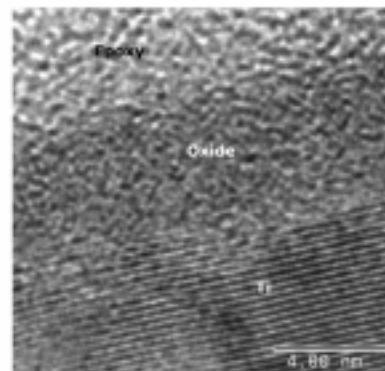


Figure 6